Enhanced thermal performance of form‑stable phase change materials with organic and inorganic supporting nanofllers

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Abstract

Organic phase change materials (PCMs) are inexpensive, safe, and do not segregate. However, they exhibit a low thermal conductivity. Several papers have reported the synthesis of organic–inorganic hybrid PCMs by adding inorganic fllers to increase the thermal conductivity. However, large amounts of fllers are needed to support the shape for the encapsulation of PCMs. Herein, we propose a facile synthesis strategy that yields composite PCMs with enhanced thermal performance. Initially, 1,3:2,4-dibenzylidene sorbitol (DBS) organogel was added as an organic fller to poly(ethylene glycol) (PEG) PCMs for maintaining the form and prevent leakage. Subsequently, a small amount of an inorganic fller, graphene nanoplatelets (GNPs), was added to these organic PCMs. Consequently, the thermal conductivity and shape stabilization of the PEG/GNPs/ DBS PCMs were signifcantly improved. These prepared composite PCMs, with excellent shape stabilization, appropriate

latent heat, and ideal thermal conductivity, are potential as fillers in solar-thermal systems and energy-efficient buildings.

Keywords Nanofbrils · Graphene nanoplatelet · Phase change material

Introduction

Recently, thermal energy storage has garnered signifcant attention because it allows the transfer and storage of thermal energy, reducing the overall energy demand. Latent heat thermal energy storage systems are the most common approaches owing to the associated high energy storage density and small temperature change during heat release; particularly, phase change materials (PCMs) are prevalently employed for latent heat thermal energy storage [\[1–](#page-7-0)[3](#page-7-1)]. PCMs release or absorb latent heat during phase change process and are used in several applications such as building energy conservation, solar thermal energy, green textiles, and lithium-ion batteries [\[4](#page-7-2)–[6\]](#page-7-3).

PCMs are divided into three types: inorganic, organic, and eutectic organic and inorganic compounds [\[7](#page-7-4), [8\]](#page-7-5). Inorganic PCMs exhibit excellent thermal conductivity but have several disadvantages including corrosion, supercooling, and lack of thermal stability. Contrastingly, organic PCMs have

 \boxtimes Wei-Chi Lai wclai@mail.tku.edu.tw no corrosive problems and do not show supercooling and phase segregation, but their thermal conductivity is low. As the drawbacks of inorganic PCMs are more critical than those of organic PCMs, the latter have attracted signifcant attention in recent years [[9–](#page-7-6)[12\]](#page-8-0). Organic PCMs involve linear long chain hydrocarbons, esters, and poly(ethylene glycol) (PEG). PEG is the most popular organic PCM [[12\]](#page-8-0) because it is non-fammable, inexpensive, and exhibits good latent heat. Therefore, PEG was chosen for this study.

The leakage of melted (liquid) PCMs leads to increased cost and thermal resistance [[9](#page-7-6)]. Recently, several papers have reported the preparation of form-stable (also called shape-stabilized) PCMs to avoid the leakage problems; many synthesizing methods have been proposed, such as encapsulation, adsorbing or soaking porous material, and blending or copolymerization of polymers as supporting materials [\[13](#page-8-1)[–16](#page-8-2)]. Belessiotis et al. investigated the preparation of shape-stabilized paraffin/ $SiO₂$ composite PCMs using a chemically aided infltration method [[13\]](#page-8-1). The PCMs with 80 mass% paraffin exhibited the highest encapsulation ratio and latent heat of 156 J g^{-1} . Hasanabadi et al. employed the vacuum impregnation method to synthesize PCMs comprising parafn/expanded perlite composites. The latent heat decreased around 18%; however, no signifcant leakage was observed [\[14\]](#page-8-3).

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The organic PCMs exhibit low thermal conductivity. Consequently, several studies have investigated the synthesis of organic–inorganic hybrid PCMs with added inorganic fllers to increase the thermal conductivity [\[17](#page-8-4)[–24\]](#page-8-5). Generally, solid particles, such as metal particles, metal oxides, carbon nanofbers, carbon nanotubes, and graphene oxide (GO) or graphene nanoplatelets (GNPs), are dispersed into organic PCMs. However, large amounts of inorganic particles are required to support the shape imparted by the encapsulation of PCMs to prevent leakage. For example, Mehrali et al. reported an evident increase in the thermal conductivity of palmitic acid (PA) PCMs using GO as the supporting material [[21](#page-8-6)]. However, approximately 50 mass% GO was required for enhancing the thermal conductivity. Lin et al. investigated the thermal properties of PA-composite PCMs for thermal energy storage. PCMs with 30 mass% $SiO₂$ demonstrated the least leakage percentage [[24](#page-8-5)]. In addition, organic supporting fllers such as high-density polyethylene, polymethyl methacrylate, cellulose diacetate, and balsa wood have also been added to prevent leakage [[25](#page-8-7)[–28](#page-8-8)]. However, the thermal conductivities of these form-stable organic PCMs are lower, limiting their practical applications.

GNPs are nanoparticles consisting of stacks of graphene sheets and exhibit good physical properties for PCM applications. In comparison with carbon nanotubes or fbers, platelet-type structures exhibit a lower thermal contact resistance at low loads, yielding high thermal conductivity. Mehrali et al. reported an enhancement of thermal conductivity of PA PCMs by the addition of GNPs, which exhibited good thermal reliability after thermal cycling [\[22\]](#page-8-9). Yang et al. found the introduction of a small amount of GNPs into PEG/boron nitride (BN) PCMs for improving the thermal conductivity; the BN content ranged between 10 and 30 mass% [\[23](#page-8-10)].

A green and cheap organogelator, 1,3:2,4-dibenzylidene-D-sorbitol (DBS), was chosen as the main supporting material for the synthesis of novel form-stable composite PCMs. DBS is derived from the natural sugarbased amphiphiles with hydroxyl groups and phenyl rings. Notably, DBS (see Fig. [1](#page-1-0)) can self-assemble into threedimensional networks with relatively small amounts in organic surroundings to produce organogels [[29–](#page-8-11)[31\]](#page-8-12). The mechanism underlying DBS network formation is controlled by the intermolecular hydrogen bonds of terminal hydroxyl groups and π interactions of phenyl rings between the DBS. The nearest hydrogen bonds and the most stable CH– π interactions are preferred for gel formation [\[29\]](#page-8-11). These DBS gel networks could signifcantly stabilize the shape by capturing melted PCMs during the phase change process. Although a few studies used organogel scafolds to keep the shape [[32](#page-8-13), [33](#page-8-14)], the thermal conductivity of PCMs was not improved by adding the organic compound DBS. Therefore, in this study, high thermal conductivity

Fig. 1 Chemical structure of DBS

inorganic fllers, GNPs, were added to increase the thermal conductivity of composite PCMs. The contribution of this study is that we demonstrate that the addition of a large amount of inorganic fllers, which are usually expensive, is not necessary to provide shape support. Alternatively, a cheap and eco-friendly sorbitol derivate, DBS, can be mainly used as the organic supporting fller to maintain the form. These DBS nanofbrillar networks capture most of the melted PCMs and stabilize the shape against leakage. To the best of our knowledge, the preparation of composite PCMs with organic and inorganic supporting nanofllers has not been investigated yet.

In the present study, DBS was added to maintain the shape of PEG PCM. The self-assembled nanofbrillar networks with DBS captured most of the PCM, stabilizing the shape to prevent the leakage during the phase transitions. Subsequently, small amounts of inorganic GNPs were added to the PEG/DBS PCMs for improving the form stabilization and thermal conductivity. The fabricated PCMs with DBS and GNPs retained its shape up to 120 °C, exhibiting excellent shape stabilization. The thermal conductivity of the PCMs was 2.3 times larger than that obtained using organic PCMs. The resulting PCMs are potential candidates for applications in solar power plants, solar water heaters, air-based heating systems, and cooling/heating of buildings, where the temperature range is approximately 40–60 \degree C [[34](#page-8-15), [35\]](#page-8-16).

Experimental

Materials

Poly (ethylene glycol) (PEG: 8000 g mole⁻¹) was obtained by Sigma-Aldrich. Graphene nanoplatelets (GNPs) were purchased from XG Sciences (Grade C: a specifc surface area of 300 m² g⁻¹). The thickness of the GNPs was approximately 1–5 nm, as provided by the supplier.

1,3:2,4-Dibenzylidene-D-sorbitol (DBS) was purchased by Milliken Chemicals.

Sample preparation

Initially, PEG (2 g) and 7 mass% DBS were mixed at 250 °C under constant agitation for 10 min. When the DBS was completely dissolved in the PEG, varying amounts of GNP (0–8 mass%) were added to the PEG/DBS solutions and maintained at 250 °C under constant agitation for 30 min. Finally, the prepared PCMs were cooled to room temperature and were maintained for more than 24 h before the experiments*.*

Dynamic Rheological test

The rheological behaviors of the composite PCMs containing 0–8 mass% GNPs were analyzed using a Physica MCR 101 rheometer (Anton Paar). The amplitude sweep test was performed over a strain range of 0.05–5% with a frequency of 10 rad s^{-1} at 25 °C. The frequency sweep test was performed between 0.1 and 800 rad s⁻¹ with a strain amplitude maintained at 0.1% at 25 °C. Gel dissolution temperatures (T_d) of the composite PCMs containing 0–8 mass% GNPs were calculated from elastic modulus *G*' versus temperature using the intersection of the tangents of the curves. The composite PCMs were heated from 80 to 150 °C with a heating rate of 5 °C min−1. The strain amplitude was maintained at 0.1%, and the frequency was 10 rad s⁻¹.

Structure

The structures of the composite PCMs containing 0–8 mass% GNPs were investigated by a H-7100 transmission electron microscope (Hitachi). The composite PCMs for TEM were prepared by adding dropwise of molten samples onto the carbon-supported TEM grids and then were maintained in a vacuum oven at 25 °C for more than 24 h. Eventually, the composite PCMs were washed by deionized water and were kept inside a vacuum oven at 40 °C for 12 h.

Heating/cooling performance

The thermal performances of the composite PCMs containing 0–8 mass% GNPs were tested through Pyris Diamond diferential scanning calorimeter (Perkin Elmer). The composite PCMs were heated from 0 to 100 °C with a heating rate of 5 $^{\circ}$ C min⁻¹, and the melting temperature (T_m) and latent heat of melting (ΔH_m) were observed. ΔH_m

can be determined from the integrating the area under the endothermic peak of the DSC curve. Subsequently, the composite PCMs were cooled from 100 to 0 °C with a cooling rate of 5 $^{\circ}$ C min⁻¹ under a nitrogen atmosphere, and the crystallization temperature (T_c) and latent heat of freezing (ΔH_c) were measured. ΔH_c was determined by integrating the area under the exothermic peak of the DSC curve.

Leakage measurement

The leakage tests of the composite PCMs containing 0–8 mass% GNPs were performed using the flter sheet sandwich method. The composite PCMs were heated to 250 °C for 5 min, and the molten PCMs were dripped onto a copper ring with a diameter of 1.5 cm and a thickness of 3 mm on a glass plate. The mass of the composite PCMs was 0.6 g. The copper ring was removed after the composite PCMs were cooled to room temperature. Subsequently, the composite PCMs were placed on a 2-mm square stainless-steel mesh (flter sheet) prior to the leakage test. The flter sheets were weighed before (M_1) and subsequent the annealing step (M_2) . The composite PCMs were weighed at diferent temperatures above the PEG melting temperature at an interval of 30 min. The leakage percentage was calculated by Eq. [\(1](#page-2-0)):

$$
\text{Leakage percentage}(\%) = \left[\frac{M_2 - M_1}{M_1}\right] \times 100\tag{1}
$$

Dimensional stability measurement

The dimensional stability measurements of the composite PCMs containing 0–8 mass% GNPs were measured using a Q400 thermomechanical analyzer (TA). The PCMs were heated from 25 to 100 °C at a heating rate of 3 °C min−1, and the constant normal force was 0.01 N.

Thermal conductivity test

The thermal conductivities of the composite PCMs containing 0–8 mass% GNPs were measured using a KD-2 Pro Model thermal properties analyzer (Decagon Devices) at 25 °C; the measurements were repeated at least thrice. The composite PCMs (12 g) were heated at 200 °C for 5 min, poured into a 7-mL vial using the SH-1 dual needle probe sensor (1.3 mm diameter, 30 mm length, and 6 mm spacing), and then cooled to 25 °C in a water bath with a temperature error within ± 2 °C for more than 24 h. The needle was completely inserted into the 7-mL vial. The measuring thermal conductivity range of the sensor was 0.02–0.2 W $m^{-1} K^{-1}$, and the read time was set to 2 min. The thermal conductivity of each composite PCM was measured at least fve times.

Fig. 2 Photographs of the composite PCMs containing 0–8 mass% GNP contents at 80 °C

Results and discussion

Phase behavior and rheological measurement

Figure [2](#page-3-0) exhibits the photographs of the composite PCMs containing $0-8$ mass% GNP contents at 80 \degree C, and is much higher than the PEG melting temperature (approximately 60 °C). A visual observation indicates the formation of transparent gels in the samples without GNPs, which may be attributed to the DBS gel network formation. The samples became translucent gels with the addition of GNPs. However, when the GNP content exceeded 8 mass%, the composite PCMs became heterogeneous, and precipitation occurred. The composite PCMs with 0–8 mass% GNPs were chosen for the subsequent investigations for the present study.

Rheometer was used to investigate the viscoelastic characteristics of the composite PCMs containing 0–8 mass% GNPs at high temperatures. Figure [3](#page-3-1) displays the (a) *G′* versus strain, and (b) *G′* and *G′′* versus frequency) of the composite PCMs containing 0–8 mass% GNPs at 80 °C. *G'* did not show signifcant variation when the strain was below 0.5% (Fig. [3](#page-3-1) a). However, it decreased signifcantly when the strain increased around 0.5%, indicating the structure started to destroy at the higher strain.

G′ was always larger than *G′′*; *G′′* and *G′′* did not signifcantly infuence with frequency (Fig. [3](#page-3-1) b), which suggests that the composite PCMs can be considered as gels. The results indicate that the masses of the PCM samples remain unchanged at 80 °C. Additionally, *G′* increased with an increase in the GNP concentration, suggesting an increase in the stifness of the composite PCMs with increasing GNP content.

Figure [4](#page-3-2) demonstrates the *G′* versus temperature of the composite PCMs containing $0-8$ mass% GNPs. T_d values were calculated from the intersection of the tangents (see dashed line in Fig. [4](#page-3-2)); the composite PCMs started to exhibit liquid states beyond T_d . The values of T_d of the composite PCMs with 0, 1, 2, 4, 6, and 8 mass% GNPs were 119.1,

Fig. 3 a *G′* versus strain, and **b** *G′* and *G′′* versus frequency) of the composite PCMs containing 0–8 mass% GNPs at 80 °C

Fig. 4 *G'* versus temperature of the composite PCMs containing 0–8 mass% GNPs

123.0, 125.9, 126.2, 126.3, and 126.3 °C, respectively. These temperatures were significantly larger than the melting temperature of PEG (approximately 60 °C). The prepared composite PCMs retain their shape until the temperature is approximately two times the melting temperature of PEG (at temperatures of up to 120° C).

Additionally, these T_d values enhanced with an increasing GNP amount. T_d exhibits a first-order transition, and it can be described by $T_{tr} (T_d) = \Delta H_{tr} / \Delta S_{tr}$, where ΔH_{tr} and ΔS_{tr} are the changes in enthalpy and entropy of the phase transi-tion, respectively [\[36\]](#page-8-17); ΔH_{tr} is the dominating parameter influencing T_d . Increasing the GNP concentration increased the thermal energy required to destroy the gels. As a result, ΔH _{tr} and T _d enhanced with increasing GNP content.

Structure

Figure [5](#page-4-0) presents the TEM micrographs of GNPs at diferent magnifcations. Multilayered sheet-shaped structures were observed, and the largest and smallest sizes of GNPs were approximately 4 μm and 500 nm, respectively. TEM micrographs of the composite PCMs containing 0, 1, and 4 mass% GNPs at diferent magnifcations are shown in Fig. [6.](#page-4-1) Threedimensional networks of DBS nanofbrils with diameters between 10 and 40 nm were observed (Fig. [6](#page-4-1) a and b), which is similar to the diameters of the fbrils in organic matrix [\[29–](#page-8-11)[31](#page-8-12)]. The fibril sizes were not significantly influenced by increasing in GNP content (Fig. [6](#page-4-1) c–f). Further, the dark regions in Fig. [6](#page-4-1) (c) and (e) indicate the varying in-plane sizes of the GNPs, and the largest sizes were approximately 4 μm. The schematic drawings of the structure of the prepared PCM are illustrated in Fig. [7](#page-4-2).

Heating and cooling performance

Figure [8](#page-5-0) demonstrates the DSC curves of the pristine PEG and composite PCMs with 0–8 mass% GNPs. All DSC experiments for each prepared sample were performed at least three times for reproducibility confrmation. The

Fig. 5 TEM micrographs of GNPs at diferent magnifcations

Fig. 6 TEM micrographs at diferent magnifcations of the composite PCMs containing **a** and **b** 0, **c** and **d** 1, and **e** and **f** 4 mass% GNPs

thermal properties of these samples are summarized in Table [1](#page-5-1). The freezing and melting temperatures of all samples were around 38 and 60 °C, respectively. These temperatures were not obviously afected with GNPs.

Contrastingly, the latent heats of freezing and melting of the composite PCMs lowered with enhancing GNP amount. For example, the highest latent heat of melting was [1](#page-5-1)85.21/J g^{-1} for the pristine PEG samples (Table 1). With the addition of DBS, the latent heat decreased to 153.44/J g^{-1} . These results suggest that the formation of

Fig. 7 Schematic drawings of the structure of the prepared PCM

Fig. 8 DSC **a** heating and **b** cooling curves of pristine PEG and composite PCMs with 0–8 mass% GNPs

DBS networks hindered the crystal structures of PEG. Similarly, for samples with varied GNP contents, the latent heat decreased with increasing GNP content. The presence of GNPs disrupted the PEG crystals, resulting in the decrease in the latent heats of the PCMs; the latent heats were above

130/J g^{-1} for the samples with 0–8 mass% GNPs. The obtained latent heat values are comparable and even higher than those reported for PEG/SiO₂ (113/J g⁻¹) and PEG/SiO₂/ Ti₄O₇ (128–139/J g⁻¹) composite PCMs [[37,](#page-8-18) [38\]](#page-8-19).

The DSC curves of composite PCMs with 8 mass% GNPs after diferent thermal cycles are presented in Fig. [9,](#page-6-0) and the resulting thermal properties are summarized in Table [2.](#page-6-1) The samples were analyzed consecutively through 10 and 50 thermal cycles from 20 to 100 °C. The phase transition temperatures and the latent heat of melting and freezing of the PCMs after 10 and 50 thermal cycles were close to those of uncycled composite PCMs. Therefore, these results suggest that the prepared PCMs demonstrated good thermal reliability.

Leakage measurement

The leakage measurements were taken at temperatures larger than the melting temperature of PEG (60 $^{\circ}$ C). Figure [10](#page-6-2) (a) exhibits the plots for leakage percentages versus temperature of the pristine PEG and composite PCMs with 0–8 mass% GNPs. The samples were weighed after maintaining the temperature above the melting temperature of PEG for 30 min. The pristine PEG samples exhibited 94% leakage at 80 °C. Contrastingly, negligible leakage was observed in the PEG/ DBS systems until 120 °C. The three-dimensional DBS networks were strong enough to immobilize melted PEG after melting. This implies the samples with DBS remained in the gel state and stabilized the form to prevent leakage. As the temperatures reached 120 °C, the DBS gel networks began to dissolve (T_d ranged from 119 to 127 °C; Fig. [4](#page-3-2)); the leakage enhanced rapidly after 120 °C.

Figure [10](#page-6-2) (b) displays the plot for leakage percentages versus time of the composite PCMs containing 0–8 mass% GNPs at 125 °C, which was close to or higher than the T_d of DBS. Consequently, at 125 °C, the PCMs exhibited the liquids, and the leakage was controlled by the GNPs. The leakage percentage of the composite PCMs with 4 and 8 mass% GNPs lowered with increasing GNP amount. The introduction of GNPs reduced the leakage and stabilized the shape owing to the hydrogen bonding and high specifc surface area of the

Table 1 Thermal data of the pristine PEG and composite PCMs with 0–8 mass% GNPs

Fig. 9 DSC curves of composite PCMs with 8 mass% GNPs after different thermal cycles

GNPs, which were advantageous for the shape-stabilized PCMs during the solid–liquid transition.

Shape stability

Figure [11](#page-7-7) displays the TMA curves of the pristine PEG and composite PCMs with 0–8 mass% GNPs. The dimensions of the pristine PEG samples lowered sharply when the temperature was heated approximately 60 °C. With a further increase in the temperature, the pristine PEG samples completely melted at approximately 65 °C. Although the thickness of the samples with DBS decreased slightly at 60 °C, the thickness of the samples with higher GNP content (4 and 8 mass%) did not change at 60 °C, indicating that the samples with DBS and GNPs retained their shape above 60 °C. These suggest the enhanced shape-stabilized phase change behaviors for the composite PCMs synthesized in this study.

Fig. 10 a Leakage percentages versus temperature of the pristine PEG and composite PCMs with 0–8 mass% GNPs and **b** leakage percentages versus time of the composite PCMs containing 0–8 mass% GNPs at 125 °C

Thermal conductivity

Figure [12](#page-7-8) presents the thermal conductivity of the composite PCMs containing 0–8 mass% GNPs. The thermal conductivity values of the composite PCMs enhanced with increasing GNP content. The thermal conductivities of the samples with 0, 1, 4, and 8 mass% GNPs were 0.21, 0.25, 0.36, and 0.48 W/m⁻¹ K⁻¹, respectively. The thermal conductivity values of the fabricated PCMs were much larger than those of the pristine PEG. The highest thermal conductivity of the fabricated PCMs was approximately 2.3 times greater than that of PCMs without GNPs. The TMA and thermal conductivity results suggest that GNPs are promising inorganic supporting materials for organic PCMs, which exhibit enhanced shape stabilization and thermal conductivity.

Fig. 11 TMA curves of the pristine PEG and composite PCMs with 0–8 mass% GNPs

Fig. 12 Thermal conductivity of the composite PCMs containing 0–8 mass% GNPs

Regarding comparisons with previous reports [[37](#page-8-18)–[39\]](#page-8-20), the thermal conductivity of the fabricated PCMs was larger than that of PEG/diatomite/CNTs $(0.15-0.29 \text{ W m}^{-1} \text{ K}^{-1})$ and was comparable to that of PEG/diatomite (0.32–0.67 W m⁻¹ K⁻¹) and PEG/SiO₂/Cu $(0.29-0.41 \text{ W m}^{-1} \text{ K}^{-1})$ PCMs. Thus, the shape-stabilized PEG PCMs with DBS and GNPs exhibited appropriate thermal conductivity, good latent heat, and excellent shape stabilization.

Conclusions

In the present study, we prepared novel form-stable composite PEG PCMs with small amounts of DBS and GNPs as the supporting materials. Rheological measurements demonstrated that the DBS gels formed after the melting temperature of PEG, implying that these composite PCMs retained their masses and shapes during the phase transition. The DBS gel dissolution temperatures ranged between 119 and 127 °C, which were larger than the PEG melting temperature. The DBS nanofibrillar network, measuring 10–40 nm in diameter, can capture the majority of the melted PCMs to prevent leakage. DSC analysis implies the latent heats of the composite PCMs were higher than $130/J.g^{-1}$. TMA results indicate the dimensions of the composite PCMs remain unchanged for the samples with DBS and GNPs, suggesting the excellent form-stable behaviors. Furthermore, the thermal conductivities of the composite PCMs increased signifcantly with increasing GNP content.

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